



U.S. Department of Energy Biofuels Program Document Database

**NREL/DOE Ethanol Pilot-Plant: Current Status
and Capabilities**

(Title)

from

Elsevier Science

(Source)

Please note that this is a copyrighted article posted by permission of the publisher. You may read it or print a copy for your personal use, but should not distribute to others and are urged to go to the original source for additional related material. This article was published by Elsevier. Their Web site is <http://www.ScienceDirect.com>.

NREL/DOE ETHANOL PILOT-PLANT: CURRENT STATUS AND CAPABILITIES

Q. A. Nguyen, J. H. Dickow, B. W. Duff, J. D. Farmer, D. A. Glassner, K. N. Ibsen, M. F. Ruth, D. J. Schell, I. B. Thompson & M. P. Tucker

Alternative Fuels Division, National Renewable Energy Laboratory, 1617 Cole Boulevard, Golden, CO 80401, USA

Abstract

The National Renewable Energy Laboratory (NREL) has built and operated a pilot-plant to convert lignocellulosic feedstocks to ethanol for the U.S. Department of Energy (DOE). The process development unit (PDU) has a designed throughput of 1 ton (dry basis)/day of biomass and is equipped to handle a variety of feedstocks. Major processing systems include feedstock milling, pretreatment, simultaneous saccharification and fermentation (SSF), and ethanol distillation. Several experimental runs have been successfully completed since the startup of the plant in March 1995. The plant capabilities are continually being improved to meet the needs of our industrial partners and to facilitate NREL's process development work. This paper reports on various aspects of commissioning and operations, the present capabilities, and plans for use of the facility. Copyright © 1997 Elsevier Science Ltd.

Key words: Biomass, bioconversion, enzyme, ethanol, fermentation, lignocellulose, pilot-plant, pretreatment, SSF.

INTRODUCTION

Pilot-plants and commercial-scale facilities for converting lignocellulosic biomass to ethanol have existed since the mid-1900s. All early plants used acids to convert or hydrolyze the cellulose to glucose, which was subsequently fermented to ethanol. Examples include the acid percolation process developed in Germany by Scholler & Associates (1937), and further refined at the U.S. Forest Products Laboratory in Madison, Wisconsin (Harris & Beglinger, 1946; Harris *et al.*, 1946), and by the Tennessee Valley Authority (Gilbert *et al.*, 1952). Other acid-hydrolysis pilot-plants have limited capability for investigating an integrated process. Typically, these plants have equipment for performing acid hydrolysis only. Examples have included plug flow reactors for continuous cellulose hydrolysis (Church & Wooldridge, 1981; Brennen *et al.*, 1987), percolation reactors (Burton, 1983), extruders (Rugg

et al., 1983; Lawford *et al.*, 1984), and paper and pulping equipment (Bulls *et al.*, 1991).

Many newer plants that investigate ethanol production from lignocellulosic biomass use enzymatic conversion technology. Again, many have limited abilities to investigate a continuous and integrated process. Early work on simultaneous saccharification and fermentation (SSF) was conducted at the University of Arkansas (Becker *et al.*, 1981). The plant used 1250-l fermenters for cellulase production and SSF, but had no pretreatment capability until a hydropulper was added to process municipal solid waste (Bevernitz *et al.*, 1982). Several newer plants have equipment for both pretreatment and fermentation, but still have limitations for whole process demonstration. Some limitations are a lack of 24-h/day processing, limited capability to handle feed materials, and a lack of distillation and solid-liquid separation equipment. Iotech (Curtin, 1983; Foody & Foody, 1991) has a pilot-plant that uses steam-explosion pretreatment, but has limited fermentation capability. A pilot-plant in Soustons, France, (Heard & Schabas, 1984) uses a Stake Technology machine for steam-explosion pretreatment and conducts fermentations in large fermenters (e.g. 30,000–50,000 l). A pilot-plant at the Voest-Alpine Biomass Technology Center used a 3000-l batch steam digester for pretreatment and produced cellulase enzyme or performed saccharification in 15,000-l fermenters (Hayn *et al.*, 1993). A smaller pilot-plant was built by Ralph Katzen Associates International with the University of Arkansas (Easley *et al.*, 1989) that performed an alkali pretreatment in a disc refiner and had a 10,000-l fermenter for the SSF process. A pilot-plant in Izumi City, Japan (Matsui, 1991) used 25-l steam-explosion vessels to produce pretreated wood that was then washed to remove inhibitors and fed to a batch 5000-l fermenter with a flash distillation system. A 500 kg/day continuous and integrated plant was built in Japan (Shirasaka *et al.*, 1989) by the Research Association for Petroleum Alternatives Development (RAPAD). This plant had unit operations for alkali pretreatment, cellulase production, saccharification, fermentation, and ethanol recovery.

The DOE Biofuels Systems Division is committed to support industry in its efforts to commercialize large-scale, cost-effective processes for producing alternative fuels, including ethanol from lignocellulosic biomass (Wyman & Hinman, 1990). Conducting bench- and pilot-scale work in concert with industrial partners is part of this commitment. Toward that end a prototype pilot-plant, or process development unit (PDU), has been installed at the National Renewable Energy Laboratory in Golden, Colorado. The pilot-plant equipment, which cost \$11.3 million, is targeted for demonstrating the enzymatic conversion of lignocellulosic biomass to ethanol based on the favorable economics of this process (Hinman *et al.*, 1992). This report summarizes the current status of the NREL pilot-plant, and reviews its initial operation and plans for use.

DESIGN CRITERIA FOR THE NREL PILOT-PLANT

The NREL pilot-plant is designed to investigate integrated processes for the enzymatic conversion of lignocellulosic biomass to ethanol. It is designed to provide scale-up data and relevant operating experience. Based on our computer process simulation (Hinman *et al.*, 1992), bench-scale experimental data, and cost considerations, we have specified the following major design criteria for the pilot-plant:

- Use the smallest scale possible that allows engineering data to be gathered for commercial plant design.

- Use enzymatic conversion technology (SSF).

- Operate continuously (24 h/day).

- Maintain a high degree of flexibility to handle a variety of feedstocks.

- Maintain the capability to handle high-solids biomass slurry (25% total solids in the fermenters).

- Use proven equipment (when possible) to ensure reliability.

- Provide BL1-LS containment capability for recombinant organisms.

- Maintain on-line data acquisition and control.

Ensure the PDU is instrumented as required to fully evaluate the results of experiments and to provide data for evaluating the feasibility of commercial plant investment.

All these criteria contribute to the unique capability offered by the NREL pilot-plant as a 'user facility' that has the capability to demonstrate fully integrated bioconversion processes, including feedstock handling, pretreatment, fermentation, distillation, solid-liquid separation, and recycle streams.

PILOT-PLANT PROCESS AND EQUIPMENT DESCRIPTION

The major equipment in the NREL pilot-plant is shown schematically in Fig. 1. The following paragraphs provide a description of the equipment and the intended process. The capability of the equipment is described in more detail in the following sections.

Feedstock handling

Wood chips are processed through a wash step to remove dirt and heavy contaminants. The washed chips are milled in a hammer mill to 1–2-mm particle size, then conveyed to the pretreatment area. Reducing the particle size eliminates potential mass and heat transfer limitations during pretreatment and fermentation processes. Grass-type feedstocks are received as bales consisting of material in long stalks that would be difficult to process without size reduction. Therefore, grasses are shredded, then sent to the wash step. Paper feedstocks are also received as bales and are shredded and sent directly to pretreatment. Paper feedstocks do not require washing because the contaminants have been removed at the wastepaper recycling plant.

Pretreatment

In the pretreatment step, the hemicellulosic fraction of the feedstock is hydrolyzed to soluble sugars (primarily xylose). This step also increases the cellulase

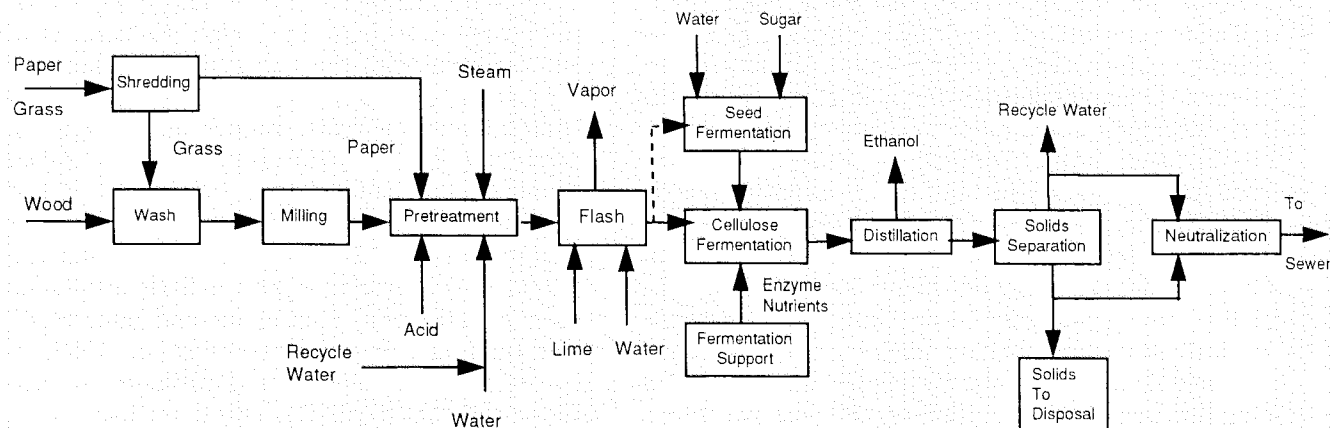


Fig. 1. NREL pilot-plant: biomass to ethanol process schematic.

enzyme's ability to convert the major fraction of the feedstock (cellulose) to soluble glucose. Following enzymatic hydrolysis of the cellulose, both sugars (glucose and xylose) can be converted to ethanol by fermentation. The pretreatment step mixes the feedstock with sulfuric acid and water (at approximately 1% acid in the resulting solution), then raises the slurry (20–25% w/w solids) to reaction temperature (160–200°C) with steam. The mixture is held at the reaction temperature for a predetermined time (2–20 min) then flashed into a tank maintained at near atmospheric pressure. Because of the sudden pressure drop, a fraction of the steam condensate and volatile compounds formed during the heating is evaporated and removed as flash tank overhead, which is condensed and sent to waste treatment. Lime is added to the remaining slurry to adjust the pH to 4.5.

Fermentation

The neutralized pretreated material from the flash tank is pumped into the 9000-l fermenters, where xylose is converted to ethanol or the pretreated material can be combined with cellulase enzyme, nutrients, and inoculum from the seed fermenters. Cellulase enzyme converts the cellulose to glucose, which is simultaneously converted to ethanol by a fermenting organism. Alternatively (as shown by the dotted line in Fig. 1), some of the pretreated material can be sent to the seed fermenters and hydrolyzed with cellulase to produce glucose for inoculum growth.

Distillation and downstream processing

After fermentation, the broth is sent to distillation. The product from the bottom of the distillation column is sent to a solids separation step. The liquid effluent can be used as recycle water for the process or sent to a neutralization step for pH adjustment before being disposed of in the city sewer system. The solids can be disposed separately or sent to the neutralization step for pH adjustment and disposal. We are installing a digester to evaluate anaerobic treatment of various waste streams from the pilot-plant. We have integrated additional pretreatment equipment to the pilot-plant to meet the needs of industrial clients.

COMMISSIONING OF THE NREL PDU

The NREL PDU was mechanically completed in August 1994. Commissioning of the plant was carried out in three major stages: (1) operational readiness review (ORR), (2) equipment performance verification, and (3) integrated equipment testing. These post-mechanical completion activities are described in the following sections.

Operational readiness review

Before starting formal operations in the PDU, a readiness verification procedure was undertaken as part of NREL's formal risk assessment policy. All facilities and operations at NREL must be systematically evaluated to identify environmental, safety, and health hazards. The organization responsible for new or revised facilities or operations must initiate a readiness verification process before occupancy or the start of new operations. The level of activity associated with the readiness review process varies with the type of risk assessment performed. Operations requiring a safe operating procedure (SOP) typically receive a basic readiness verification, and facilities or operations that require a safety analysis review (SAR) (Duff *et al.*, 1994) receive a formal ORR. The ORR is the highest level of readiness verification.

The magnitude of the new PDU installation and the proposed uses of the new facility require that both a SAR and a hazard and operability study (HAZOPS) be conducted. The SAR and HAZOPS both took place during the design phase of the project. These studies identified, evaluated, and proposed controls for the risks associated with installing and operating the PDU. Based on the NREL risk assessment policy, the SAR and HAZOPS dictated that a formal ORR was required. The purpose of the ORR was to verify that all administrative, procedural, and engineering controls identified and required by the SAR and HAZOPS processes were in place.

To accomplish the ORR, an ORR panel was established that comprised representatives from the NREL Environment, Safety and Health Office, Facilities, and the Bioprocess Development team. This seven member panel established the ORR plan, which required separating the PDU process into individual systems and developing comprehensive ORR checklists for each system. ORR checklists were also developed for procedural and administrative systems to ensure these non-engineering controls were in place. Twenty-two ORR checklists were developed: 11 process systems, two utility systems, and nine administrative systems (Table 1). Once approved by the ORR panel, the checklists were provided back to the PDU startup teams to facilitate self-assessment before the formal ORR.

As each PDU process or administrative system was completed, the startup teams notified the ORR panel they were ready for formal evaluation of the ORR checklists. At that point, individual ORR teams made up of staff from throughout NREL performed a preliminary ORR of each system using the approved checklists. Following the preliminary ORR, any open items on the system's checklist had to be corrected or resolved. When the open items were corrected, the final ORR was scheduled to verify that the open items were indeed closed out.

Table 1. List of ORR checklists for the NREL pilot-plant

| No. | Description of the system |
|-----|---|
| 1 | Wash system |
| 2 | Milling and conveying system |
| 3 | Pretreatment system |
| 4 | Fermentation support system |
| 5 | 160-l Fermenter system |
| 6 | 1450-l Fermenter system |
| 7 | 9000-l Fermenter system |
| 8 | Distillation and ethanol storage system |
| 9 | Neutralization system |
| 10 | Centrifugation system |
| 11 | Recycle water system |
| 12 | Utility systems |
| 13 | Emergency preparedness |
| 14 | Fire protection |
| 15 | Chemical hazard communication |
| 16 | Confined space program |
| 17 | Radiation level gauges |
| 18 | Electrical safety |
| 19 | Environmental control |
| 20 | Hoisting, rigging, and conveyors |
| 21 | Bio-safety |
| 22 | Administrative controls, training |

The final ORR checklists were then submitted to the ORR panel.

In October 1994, based on the successful closing out of 16 of the 22 checklists, the Alternative Fuels Division Director granted approval to start operations of the verified systems. The remaining systems were closed out over the next several months, and the unconditional approval to start operations was applied for. This was granted, completing the readiness verification process and allowing unlimited operation of the facility.

Equipment performance verification

Following the ORR, each system was tested to verify its performance. The performance verification of equipment included:

- Checking the function of all instrumentation and control.

- Verifying connection and transfer between the control room and local control panel.

- Operating the equipment with selected materials.

These single-system functional tests were usually performed as soon as the ORR was complete. In this way, commissioning activities could start before the whole plant was mechanically complete. The capability and performance of several systems are described below.

Wash, milling, and conveying systems

The wash system is used to remove tramp metal, rocks, and other debris from wood chip and herbaceous feedstocks. The wash tank is a modified hydropulper, consisting of an agitated conical-shaped tank. The tank is filled with water, then agitated. Wood chips or shredded straw are dumped

into the wash tank. The agitation causes the light biomass material to remain suspended in water. Heavy materials such as rocks and metal sink to the bottom and are removed separately. The washed wood chips are transferred through a side port into stainless-steel wire-mesh totes. The wash tank can process a 200 kg (dry weight) batch of wood chips per wash. We find the wash system removes large, heavy debris quite well. In several tests, rocks (having a size between 3 and 4 cm) and steel bolts and nuts (less than 2 cm) were deliberately mixed with the incoming wood chips to see how well the washer separated this debris from the wood chips. The large rocks were consistently separated from the chips and recovered from the bottom of the washer. However, the small steel nuts (less than 1 cm in size) were not always removed.

The feedstock milling and conveying system consists of a tote dumper and stainless-steel totes, feed hopper, weight belts, hammer mill, pneumatic feed transfer system with baghouse to contain dust, vibratory screen separator, and a cleated belt conveyor. This system reduces the particle size of biomass material, meters, and measures the weight of material being fed continuously into the pretreatment equipment. The particle size of the milled feedstock can be controlled by changing the screen size of the vibratory separator. Milled material is dumped into the feed hopper, which is equipped with a screen with 6-mm openings to prevent debris from entering the pretreatment system. The material is metered from the bottom of the feed hopper onto a weight belt then transferred by the cleated belt conveyor into the plug mill mixer.

Pretreatment system

The pretreatment system consists of a pug mill mixer, cross-feeder, and plug flow feeder feeding into a Sands Hydrolyzer pretreatment reactor followed by a flash tank and a hydrolyzate pump. In addition, a squeeze pump is used to recycle squeeze from the plug flow feeder into the vertical impregnator section of the Sands Hydrolyzer for preimpregnation of the feedstock with dilute acid. The 316 stainless-steel plug mill mixer is used to mix acid and water to arrive at the desired acid/feedstock ratio (pH) and solid concentration within the Sands Hydrolyzer (Sands Defibrator, Inc., USA). The plug flow feeder compresses the acid-wetted feedstock into a plug solid enough to resist the steam pressure (maximum 400 psig) within the Sands Hydrolyzer. Heating of the acid-impregnated feedstock is by direct steam injection. The residence time of material within the Sands can be varied over a considerable range. Double reciprocating valves isolate the Sands from the flash tank and allow the pretreated biomass to be metered via changes in the rate of valve cycling. The hydrolyzate pump meters the pretreated and flash-cooled biomass into the

9000-l fermenters. Lime and additional water may be added and mixed at the flash tank to arrive at the desired pH and solids concentration of feed to the fermenters.

We have successfully tested the pretreatment system with wood chips, sawdust, and corn fiber. We have solved several challenging problems related to pumping slurries of pretreated lignocellulosic material. Progressive cavity pumps work well with extensively pretreated materials (i.e. very fine solid particles with fiber length less than 1 mm) at low-solids concentrations. Fibrous materials (with fiber length greater than 1 mm) tend to cause the progressive cavity pumps to bind, dewater the slurry, and eventually fail. Open-faced centrifugal pumps and rotary lobe pumps perform well on high-solids slurries of fibrous material (fiber length of up to 2 mm). Sterility and ease of sterilization are other important criteria for selecting hydrolyzate pumps. Progressive cavity pumps are difficult to sterilize adequately using steam.

Fermentation systems

The fermentation systems include the seed train (which consists of a 20-l New Brunswick Bioflo IV fermenter, two 160-l fermenters, and two 1450-l fermenters), the fermentation support system, and four 9000-l SSF fermenters (Associated Bio-engineers and Consultants, Inc., USA) connected in series. The seed train generates inoculum for the SSF fermenters. The fermenting organism is grown from shake flasks, then transferred, in sequence, to the larger fermenters in the seed train. The fermentation support vessels hold enzymes, nutrients, and inoculum generated from the seed train.

Cellulose conversion is carried out in the 9000-l fermenters. Pretreated biomass slurry, enzyme, inoculum, and nutrient are metered into the first 9000-l fermenter. Each 9000-l fermenter has a hydraulic retention time of 24 h. However, the residence time can be changed by varying the level in the fermenters. Satisfactory level control has been achieved by using load cells. To date, we have operated the SSF fermenters at solids loadings as high as 15 wt% fibrous material without any difficulties.

Distillation

The distillation system, supplied as a complete package by APV Crepaco, Inc. (USA), consists of a 304 stainless-steel column, a steam sparger, two 100%-duty feed vertical shell and tube preheaters, one watercooled shell and tube product condenser, one chilled-water-cooled shell and tube vent condenser, bottoms and reflux pumps, a reflux tank, a bottoms cooler, associated piping and instrumentation. The distillation column, 16 in. diameter \times 31 ft long, has 14 stripping sieve trays (with 3/8 in. holes), five rectifying valve trays, and 14 cleanout ports. The

steam sparger was selected instead of reboiler heat exchangers to minimize the anticipated scaling problem as a result of using lime for pH adjustment of pretreated material. The system was designed to strip ethanol from the fermentation broth, concentrate the distillate to 100 proof (50% by volume), and leave less than 100 ppm ethanol in the bottoms. The column, which was designed for a feed flow rate of 4 gallons per minute (GPM), has been successfully operated at feed rates up to 6 GPM.

The installed distillation system contains well-known technology, so little future design work will be done for it. However, future designs will have to account for gypsum plating-out of the broth. Gypsum is produced during neutralization of sulfuric acid with lime in the pretreatment flash tank. Gypsum becomes less soluble as the aqueous mixture is heated, so it is expected to plate-out on the heat exchangers' tubes and on the column's trays. The pressure drop across the preheaters and the column will be measured and recorded to identify the speed and amount of gypsum build-up on the equipment. The distillation system uses two 100%-duty feed preheaters so that one can be taken off line for cleaning while the system is in operation. A sulfamic acid clean-in-place (CIP) system has been designed to remove the plated-out gypsum from the preheater not on line. The ethanol produced from the NREL pilot-plant will be further processed into fuel by a local fuel ethanol producer. To date we have only operated the distillation for a short time, and have experienced no significant fouling problems.

Centrifugation/recycle

A 316 stainless-steel, 14-in. diameter solid bowl decanter centrifuge, Sharples Model P-3000 (England), separates the distillation bottoms slurry to a cake for disposal and a centrate for use as recycled process water. The centrifuge, which has a maximum bowl speed of 4000 rpm, has been run with a feed flow of 2–3 GPM. In one test run using enzymatically digested corn fiber, the centrifuge separated a slurry containing 6% w/w insoluble solids into a cake with 25% w/w insoluble solids and a centrate with 2% w/w insoluble solids. The insoluble solids recovered in the cake were approximately 70% of the feed. We expect higher solids recovery can be achieved with minor modifications to the cake discharge mechanism, as recommended by the centrifuge manufacturer. The centrate stream is designed to be diluted with fresh water in a user-specified proportion and recycled to pretreatment and neutralization. The dilution with water also reduces inhibitor build-up in the system. The first recycle tank sterilizes the centrate batchwise. The second holds the sterile centrate and feeds it to the process continuously. The recycle tanks sterilization cycle has been tested, but the integration of the

tanks and their capability to maintain sterility has not.

Integrated equipment testing

Following the performance verification of individual process systems, several integrated systems were tested. Initial tests involved a few systems to facilitate process and equipment trouble-shooting. More process systems were added to later tests until all systems in the pilot-plant were integrated. Each test generally lasted 4–7 days. These tests and the individual equipment performance tests also provided opportunities for training operators. During late March and into early May of 1995 two integrated equipment shakedown experimental runs were made using corn fiber. These runs were conducted to test equipment and instrumentation readiness for controlling process variables and for testing our material balance data acquisition. As a result of

these tests, several deficiencies in instrumentation and equipment were identified and corrected.

EXPERIMENTAL ACCOMPLISHMENTS AND PLANS

Experimental accomplishments

An important capability of the pilot-plant is to provide information on material balances around key unit operations such as pretreatment and fermentation. This information is crucial for designing larger-scale plants. Bench-scale data are frequently inadequate because larger-scale processes cannot be simulated with bench-scale apparatus. For example, flash vapor from the pretreatment step contains a large fraction of the total furfural produced by the degradation of xylose. The amount of furfural (a fermentation inhibitor) removed with the flash vapor is important process information. This information

Table 2. Material balance for the pretreatment of yellow poplar sawdust using the Sunds Hydrolyzer

| Conditions | | | | | | | | |
|---|-------------------------|---------------------|------------------------------|----------------|-------------|-------------|----------|-------------|
| Temperature | 160°C | Feed rate (wet wt) | 98 kg/h (wet), 54 kg/h (dry) | | | | | |
| Time | 10 min | Acid flow rate | 35 kg/h | | | | | |
| Sulfuric acid conc. in hydrolyzer | 1% | Water to plug mill | 42 kg/h | | | | | |
| Acid conc. in feed tank | 5% | Steam rate | 45 kg/h | | | | | |
| Solid conc. in hydrolyzer | 24.5% | Water to flash tank | 22 kg/h | | | | | |
| Sawdust solid conc. | 55% | Flash vapor | 21 kg/h | | | | | |
| Results | | | | | | | | |
| Solids solubilized | 31% | | | | | | | |
| Monomer/total sugar ratio of water solubles | | | | | | | | |
| Glucose | 98% | | | | | | | |
| Mannose | 89% | | | | | | | |
| Xylose | 100% | | | | | | | |
| Component | Unpretreated (% dry wt) | Pre-treated | | | | | | |
| | | In solids | | In liquid | | | In flash | |
| | | (% dry wt) | (% in feed) | (g/l) monomers | (g/l) total | (% in feed) | (g/l) | (% in feed) |
| Glucose | 54.4 | 64.1 | (97) | 8.6 | 8.8 | (6.7) | | |
| Mannose | 5.9 | 0 | | 7.0 | 7.9 | (46.5) | | |
| Galactose | 0 | 0 | | 3.4 | 3.9 | (100) | | |
| Xylose | 17.8 | 3.1 | (12) | 39.3 | 38.6 | (75.4) | | |
| Arabinose | 0 | 0 | | 1.9 | 2.3 | (100) | | |
| Lignin | 26.5 | 33.8 | (88) | | 4.3 | (5.6) | | |
| Acetic acid | | | | | 13.1 | | 0.5 | |
| Furfural | | | | | 1.5 | (4.6) | 18.6 | (6.3) |
| HMF | | | | | 0 | | 0 | |

cannot be accurately obtained by small-scale pretreatment and reaction bomb experiments. Likewise, fermentation exhaust gas flow rates and compositions are easier to measure at a larger scale.

Mass balances are calculated from a variety of sensor readings (e.g. flow rates, temperatures, etc.) and analytical data. Material balances on larger-scale continuous processes are more difficult than bench-scale data because they require numerous sensor readings. Six flow rate and two temperature measurements, and typical analytical data (i.e. solids content and liquid and solid compositions of untreated and pretreated feedstock) are required in the NREL pilot-plant to calculate mass balances around the pretreatment system. However, the results from the pilot-plant have been similar to bench-scale results where available, and confirm our ability to close mass balances.

The material balance for a pretreatment run using yellow poplar sawdust is listed in Table 2. The clean sawdust, obtained from a sawmill in West Virginia, did not require washing. In this run, the steam consumption was approximately 0.8 kg per kg of dry wood, and 46% of the condensate flashed off in the flash tank. The flash vapor contained about 60% of the total furfural generated. However, most of the acetic acid remained in the slurry. The mass balance closure for glucose was 100%, xylose 92%, and lignin 93%. The pretreatment equipment has been operated several times for experimental determination of optimal conditions for integration with fermentation processes. This is an on-going project.

Several equipment capabilities have been added since mechanical completion. Industrial partner equipment has been installed in the pilot-plant. This was necessary but has delayed the scheduled experimental activities. Additionally, the fermenters and other stainless-steel vessels were passivated with nitric acid to protect them against corrosion. Planned utility upgrades for cooling water and compressed air have also been completed.

Future plans

The pretreatment equipment is being integrated with intermediate-scale fermentation equipment housed outside the pilot-plant so continuous feed can be provided to a smaller-scale process development effort. This system is used to investigate processes before using the PDU. It has advantages over bench-scale systems in that the biomass slurries can be better handled. However, it is not capable of handling the very high solid concentration slurries used in the PDU.

The capabilities of the PDU will continue to be enhanced to meet the needs of industrial partners. New equipment will also be added to test new processes that may lower the cost of ethanol production from lignocellulosic biomass.

Aspen, yellow poplar, corn fiber, rice straw and mixed wastepaper have been tested in the pilot-plant

equipment to date. So far, we have tested a variety of material handling equipment (such as conveyor, mill for size reduction, and pump) and determined the operating parameters most suitable for processing these biomass materials. Experimental plans will include work on corn fiber, rice straw, and yellow poplar with the current and new industrial partners.

The NREL pilot-plant is beginning to have an impact on the evaluation of processes for converting lignocellulosic materials to ethanol. Shakedown experiments are complete. Current and future NREL partners will use the pilot-plant to continually evaluate processes and technology improvements. The pilot-plant's role in commercializing biomass to ethanol technology is just beginning.

ACKNOWLEDGEMENT

This work was funded by the Biochemical Conversion Element of the Office of Fuels Development of the U.S. Department of Energy.

REFERENCES

- Becker, D. K., Blotkamp, P. J. & Emert G. H. (1981). Pilot-scale conversion of cellulose to ethanol. *Fuels from Biomass and Waste*. Ann Arbor, Michigan, pp. 375-391.
- Bevernitz, K. J., Gracheck, S. J., Rivers, D. B., Becker, D. K., Kaupisch, K. F. & Emert, G. H. (1982). Development of enzyme-catalyzed cellulose hydrolysis process for ethanol production. *Energy From Biomass and Waste VI*. Institute of Gas Technology, Chicago, pp. 897-918.
- Brennen, A. H., Hoagland, W. & Schell, D. J. (1987). High temperature acid hydrolysis of biomass using an engineering-scale plug flow reactor: results of low solids testing. *Biotechnol. Bioengng Symp. No. 17*, pp. 53-70.
- Bulls, M. M., Watson, J. R., Lambert, R. O. & Barrier, J. R. (1991). Conversion of cellulosic feedstocks to ethanol and other chemicals. *Energy from Biomass and Waste XIV*. Institute of Gas Technology, Chicago, pp. 1167-1179.
- Burton, R. J. (1983). The New Zealand wood hydrolysis process. *Proc. Royal Society of Canada Int. Symp. on Ethanol from Biomass*, Ottawa, Canada. Royal Society of Canada, pp. 247-270.
- Church, J. A. & Wooldridge, D. (1981). Continuous high-solids acid hydrolysis of biomass in a 1.5 in. plug flow reactor. *Ind. Engng Chem. Res. Dev.*, **20**(2), 371-378.
- Curtin, M. E. (1983). Canadian wood-to-ethanol projects enter pilot stage. *Biotechnol.*, **1**(2), 139-140.
- Duff, B. W., Werdene P. & Shannon P. (1994). Safety analysis report — process demonstration unit. NREL/MP-190-71 17.
- Easley, C. E., Bevernitz, K., Becker, D., Stewart, E. A. & Hetzel, E. F. (1989). Cellulosic waste conversion to ethanol using fed batch, simultaneous saccharification and fermentation. *Energy from Biomass and Waste XIII*, Institute of Gas Technology, Chicago, pp. 1295-1310.
- Foody, B. E. & Foody, K. J. (1991). Development of an integrated system for producing ethanol from biomass. *Energy from Biomass and Waste XIV*, Institute of Gas Technology, Chicago, pp. 1225-1243.

- Gilbert, N., Hobbs, I. A. & Levine, J. D. (1952). Hydrolysis of wood using dilute sulfuric acid. *Ind. Engng Chem.*, **44**, 1712–1720.
- Harris, E. E. & Beglinger, E. (1946). Madison wood sugar process. *Ind. Engng Chem.*, **38**, 890–895.
- Harris, E. E., Hajny, G. J., Hannan, M. & Rogers, C. (1946). Fermentation of douglas fir hydrolyzate by *S. cerevisiae*. *Ind. Engng Chem.*, **38**, 896–904.
- Hayn, M., Steiner, W., Klinger, R., Steinmüller, H., Sinner, M. & Esterbauer, H. (1993). Basic research and pilot studies on the enzymatic conversion of lignocelluloses. In *Bioconversion of Forest and Agricultural Plant Residues*. CAB International, Wallingford, UK, pp. 33–72.
- Heard, J. & Schabas, W. (1984). France puts emphasis on biomass conversion plan. *Chem. Engng*, **91**(6), 49–51.
- Hinman, N. D., Schell, D. J., Riley, C. J., Bergeron, P. W. & Walter, P. J. (1992). Preliminary estimate of the cost of ethanol production for SSF technology. *Appl. Biochem. Biotechnol.*, **34/35**, 639–650.
- Lawford, R., Charley, R., Edamura, R., Fein, J., Hopkins, K., Potts, D., Zawadzki, B. & Lawford, H. (1984). Biomass to ethanol by the bio-hol process. *Fifth Canadian Bioenergy R and D Seminar*, National Research Council of Canada, Ottawa, Canada, pp. 503–508.
- Matsui, S. (1991). Development of fuel alcohol technologies: research and development of a total system—total system using woody biomass. *Eleventh Annual Conf. on Alcohol and Biomass Energy Technologies*, NEDO-OS-9106, New Energy and Industrial Technology Development Organization, Tokyo, pp. 27–40.
- Rugg, B., Armstrong, P., Dreiblatt, A. & Wise, D. L. (1983). Liquid fuel and chemicals from cellulosic residues by acid hydrolysis. *Liquid Fuel Developments*. CRC Press, Boca Raton, Florida, pp. 139–158.
- H. Scholler & Associates (1937). U.S. Patents 2,083,347 and 2,083,348.
- Shirasaka, Y., Ishibashi, H., Etoh, H., Michiki, H., Miyakawa, H. & Moriyama, S. (1989). An integrated ethanol process based on advanced enzyme, fermentation, and ethanol recovery technologies. *Energy from Biomass and Waste XIII*, Institute of Gas Technology, Chicago, pp. 1311–1327.
- Wyman, C. E. & Hinman, N. D. (1990). Ethanol: fundamentals of production from renewable feedstocks and use as a transportation fuel. *Appl. Biochem. Biotechnol.*, **24/25**, 735–753.

Dr Howard Brown
Mail Stop 3511
Biofuels/Biotechnology Communications
National Renewable Energy Laboratory
1617 Cole Boulevard
Golden, CO 80401-3393
USA

Dear Dr Brown

BIORESOURCE TECHNOLOGY, vol 58, 1996, pp 189-196, "NREL/DOE Ethanol Pilot-Plant..."

As per your letter dated 12 December 2002, we hereby grant you permission to reproduce the aforementioned material on the NREL web site at no charge subject to the following conditions:

1. If any part of the material to be used (for example, figures) has appeared in our publication with credit or acknowledgement to another source, permission must also be sought from that source. If such permission is not obtained then that material may not be included in your publication/copies.

2. Suitable acknowledgment to the source must be made, either as a footnote or in a reference list at the end of your publication, as follows:

"Reprinted from Publication title, Vol number, Author(s), Title of article, Pages No., Copyright (Year), with permission from Elsevier Science".

3. A hypertext link is to be made to the Bioresource Technology Home Page at <http://www.elsevier.com/locate/biortech> and the ScienceDirect Home Page at <http://www.ScienceDirect.com>.

4. A notice is prominently displayed on your Web site saying that single copies of the article can be downloaded and printed for the reader's personal research and study.

5. Reproduction of this material is confined to the purpose for which permission is hereby given and excludes use in any electronic form other than on the World Wide Web as specified above.

6. This permission is granted for non-exclusive world English rights only. For other languages please reapply separately for each one required.

7. This permission is for THREE years only. After this period permission continues for posting of the abstract only.

Yours sincerely

Helen Wilson on behalf of
Frances Rothwell (Mrs)
Global Rights Manager